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A Study of High Temperature Deformation in Dispersion Strengthened Metal Single Crystals

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A STUDY OF HIGH TEMPERATURE DEFORMATION
IN DISPERSION STRENGTHENED METAL
SINGLE CRYSTALS

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20. efforts to understand their properties. More specifically our recent objectives have been to obtain and prepare dispersion strengthened metal single crystals and to design and construct apparatus for assessing their high temperature mechanical properties.

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I. SUMMARY OF RESEARCH

The objective of the work of this research program was to obtain an understanding of the mechanisms that control high temperature deformation in metal single crystals containing non-deformable particles, in the hope that such information will aid in understanding the complex mechanical behavior of commercial, polycrystalline, dispersion strengthened alloys. This objective was motivated by the belief that the presence of grain boundaries invariably degrades the creep and rupture properties of commercial dispersion strengthened metals and confuses efforts to understand their properties. We believe our work has made it easier to understand the unusual properties of dispersion strengthened metals. We hope that the design and development of dispersion strengthened metals will be facilitated by the ideas we have developed.

The main thrust of our experimental work has been on single crystals of Ni-20Cr-2ThO₂. By studying the creep properties of this material we have been able to develop a very simple and rational explanation of the unusual properties of dispersion strengthened metals. In particular, our work has indicated that the very high activation energies and stress exponents that have been reported for these materials have a direct and natural explanation. Also, the unusual and sometimes unpredictable properties of polycrystalline dispersion strengthened metals can now be understood as a result of our work on single crystals. This work has been published: R. W. Lund and W. D. Nix, "High Temperature Creep of Ni-20Cr-2ThO₂ Single Crystals", Acta Met. 24, 469 (1976). Copies of this paper are attached as requested.

The principal finding of this research has been that, for all practical purpose, the Orowan passing stress controls the high temperature

creep properties. Our experiments show that measureable creep does not occur below a critical stress close to the Orowan stress. Furthermore, we find that the creep rate above the critical stress is precisely what one would expect for the matrix in question (Ni-20Cr) if the difference between the applied stress and the critical stress is regarded as the driving force for creep. This simple model describes the creep rate to within a factor of 3 at all stresses and temperatures. It follows from the idea of the critical stress that the stress exponent will be large and variable. When the stress is just slightly above the critical stress the creep rate is extremely stress sensitive because a small variation in the applied stress might correspond to an enormous change in the effective stress. In this way we have shown that the stress exponents for creep of dispersion strengthened metals can have virtually any value, provided that the applied stress is judiciously fixed with respect to the critical stress.

Since the creep rate in dispersion strengthened metals depends very sensitively on stress, it follows that the apparent activation energy can be anomalously large. This occurs because creep deformation depends on the ratio of the stress to the elastic modulus; a large stress dependence implies a correspondingly large dependence on elastic modulus. Since the elastic modulus is temperature dependent, it follows that the creep rate is temperature dependent not only through the activation energy but also through the temperature dependent elastic modulus. We have shown that the high activation energies that have been reported for dispersion strengthened metals result from the temperature dependence of the elastic modulus. When a correction for this temperature dependence is made the activation energy falls very close to that for self diffusion, just as it does for pure metals

and solid solutions. In the present work on Ni-20Cr-2ThO₂ single crystals we also find the creep activation energy to be that for self diffusion. This work has been published: R. W. Lund and W. D. Nix, "On High Creep Activation Energies for Dispersion Strengthened Metals", Mec. Trans. 6A, 1329 (1975). Copies of this paper are attached as requested.

Our experiments on single crystals help to explain the unusual properties that have been observed for polycrystalline dispersion strengthened metals. Polycrystals creep at applied stresses that are well below the Orowan stress. This is the result of grain boundary sliding and the development of stress concentrations at grain boundary junctions. At points of stress concentration the Orowan stress is exceeded and creep occurs. Elsewhere in the sample the Orowan stress or critical stress is not exceeded and creep does not occur. Fracture evidently occurs before the entire sample can undergo creep deformation. Thus creep in polycrystalline samples is a highly inhomogeneous process. In single crystals the deformation occurs more homogeneously with the consequence that the basic mechanisms are more clearly revealed.

As noted above, our work on the creep properties of Ni-20Cr-2ThO₂ indicated a critical threshold stress below which creep could not be detected. Our early work indicated that the critical stress is close to the Orowan bowing stress for our crystals. We made this determination by comparing the measured critical stress with that calculated on the basis of the observed ThO₂ particle spacing. However, such calculations always involve certain unknowns so that we cannot pinpoint the Orowan stress exactly. In an effort to strengthen this part of our work we measured the room temperature yield strength of oriented crystals of Ni-20Cr-ThO₂ and obtained the Orowan stress experimentally. The results indicated that although

the creep threshold stress is close to the Orowan stress as we had deduced earlier, in fact the threshold stress lies somewhat below the Orowan stress (the threshold stress is about 65% of the Orowan stress). This result is important because it indicates that we have, indeed, detected measureable creep below the Orowan stress. This is expected theoretically. This refinement of our understanding of the threshold stress does not alter our general finding that the Orowan stress controls virtually all of the creep properties of dispersion strengthened crystals. This work has been accepted for publication: G. M. Pharr and W. D. Nix, "A Comparison of the Orowan Stress with the Threshold Stress for Creep for Ni-20Cr-2ThO₂ Single Crystals" (to be published in Scripta Met.) a copy of the preprint is attached as requested.

Our study has indicated that dispersion strengthened single crystals are inherently very ductile. While polycrystalline dispersion strengthened metals typically fail in creep after about one percent strain, our single crystals showed very much higher ductility. We find elongations to failure as high as 30% and reduction of areas typically in the 60-80% range. These ductilities are achievable because high angle grain boundaries are not present to cause fracture. Failure in these crystals occurs by the onset of plastic instability and necking. We have made a theoretical study of this kind of failure process. It indicates that the elongation to failure should be a very sensitive function of the apparent stress exponent for creep. In terms of our present understanding of creep in dispersion strengthened crystals the prediction is that the creep elongation to failure becomes very small when the applied stress is only slightly greater than the critical threshold stress. This prediction is verified

by the experimental results. This theoretical work has been published: M. A. Burke and W. D. Nix, "Plastic Instabilities in Tension Creep", Acta Met. 23, 793 (1975). Copies of this paper are attached as requested.

We have always been interested in the physical mechanisms by which creep occurs in dispersion strengthened metals. Our first theoretical effort was to study the rate at which edge dislocations climb over circular inclusions. Though the geometry of this problem is somewhat artificial, this study laid the ground work for our later theoretical analyses. Our first study did indicate that the elastic properties of the dispersed phase are not likely to be important to creep in dispersion strengthened crystals. This work has been published: J. H. Holbrook and W. D. Nix, "Edge Dislocation Climb Over Non-Deformable Circular Inclusions", Met. Trans. 5, 1033 (1974). Copies of this paper are attached as requested.

A more complete theoretical study of dislocation over non-deformable inclusions has been completed. The basic thrust of our study has been to determine why creep occurs so slowly below the Orowan Stress. We have made a completely new analysis of this problem taking into account the configuration of the dislocation as it climbs over the particle. This analysis indicates that the creep rate should drop rapidly below the Orowan stress, in agreement with our observations. We have also shown that the stacking fault energy may influence the creep rate by making it impossible for some dislocations to climb over particles. Considering all of these factors, we have been able to develop a quantitative description of creep below the Orowan stress that is consistent with our experiments. This work is now completed and is described in John Holbrook's PhD Thesis. Journal articles describing this work are now in preparation.

Our study of the creep properties of dispersion strengthened single crystals has raised a number of questions about the mechanisms responsible

for creep flow in particle strengthened systems. In an effort to answer these questions and provide a fuller understanding of creep in these materials we have undertaken a transmission electron microscopy study of the creep substructure in Ni-20Cr-2ThO₂. To our surprise we find the dislocation density and subgrain size to be almost exactly what one would expect for a pure metals or simple solid solution. The dispersion that so dramatically improves the strength has virtually no effect on the substructure. We are presently using this information as a basis for constructing a more complete and detailed theoretical picture creep in dispersion strengthened metals. Our transmission electron microscopy work is now complete. A paper has been prepared for publication and will be submitted to Acta Met. this month.

During the last year of this program the orientation of our work has been changing. We are now engaged in a study of the processing of dispersion strengthened metals. We are interested in mechanical alloying as well as in the thermo-mechanical processing needed to produce elongated grain structures. We have developed the technique for mechanical alloying and have reproduced certain classical experiments. We have also succeeded in mechanically alloying aluminum powder that may contain a fine dispersion of Al₂O₃. This work was described in our recent proposal to AFOSR. We have also succeeded in making elongated grain structures in an Al-Si alloy. We expect to be able to understand the factors that lead to elongated grain structures by studying this and other simple systems.

II. PUBLICATIONS, REPORTS AND DISSERTATIONS RELATING TO GRANT AFOSR-73 - 2434

A. Publications

1. J. H. Holbrook and W. D. Nix, "Edge Dislocation Climb over Non-Deformable Circular Inclusions" Metall. Trans., 5, (1974)1033.
2. M. Vikram Rao and W. D. Nix, "Creep in Binary Solid Solutions: A Possible Explanation for Anomalous Behavior" Scripta Met., 7 (1973) 1255.
3. M.A. Burke and W. D. Nix, "Plastic Instabilities in Tension Creep", Acta Met., 23 (1975) 193.
4. R. W. Lund and W. D. Nix, "On High Creep Activation Energies for Dispersion Strengthened Metals", Metall. Trans., 6A (1975), 1329.
5. R. W. Lund and W. D. Nix, "High Temperature Creep of Ni-20Cr-2ThO₂ Single Crystals", Acta Met., 24 (1976) 469.
6. G. M. Pharr and W. D. Nix, "A Comparison of the Orowan Stress with the Threshold Stress for Creep for Ni-20Cr-2ThO₂ Single Crystals" (to be published in Scripta Met.).

B. Ph.D. Dissertations

1. R. W. Lund, "A Study of High Temperature Creep of Dispersion Strengthened Ni and Ni-20Cr" Ph.D. Dissertation, Stanford University (1975).
2. J. H. Holbrook, "A Theoretical Investigation of Creep of Dispersion Strengthened Crystals", Ph.D. Dissertation, Stanford University (1976).

C. Oral Presentations (speaker underlined).

1. W. D. Nix, "On the Existence of Steady State Creep," Department of Mechanical Engineering, University of Colorado, Boulder, Colorado (December, 1973).
2. W. D. Nix, "On the Existence of Steady State Creep," Department of Metallurgical Engineering, Colorado School of Mines, Golden, Colorado (December, 1973).
3. W. D. Nix, "Plastic Instabilities in Tension Creep" Air Force Conference on: Fracture and Fatigue of Two Phase Materials - Effects of Plastic Instability Fairborn, Ohio (September, 1974).
4. W. D. Nix, "High Temperature Creep of Dispersion Hardened Single Crystals" TMS-AIME (invited paper) University of Toronto (May, 1975).

III. PROFESSIONAL PERSONNEL

The following personnel of the Department of Materials Science and Engineering at Stanford have been engaged in this research program:

Principal Investigator:

Dr. William D. Nix, Professor

Research Associate:

Dr. Jürgen Hausselt, Ph.D. University of Erlangen, Germany

Graduate Research Assistants:

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M.S., Stanford University

Mr. Richard W. Lund, B.S., M.S., University of Wisconsin

Mr. Paul S. Gilman, B.S., University of Pennsylvania
M.S., Stanford University

Mr. George M. Pharr, B.S. Rice University